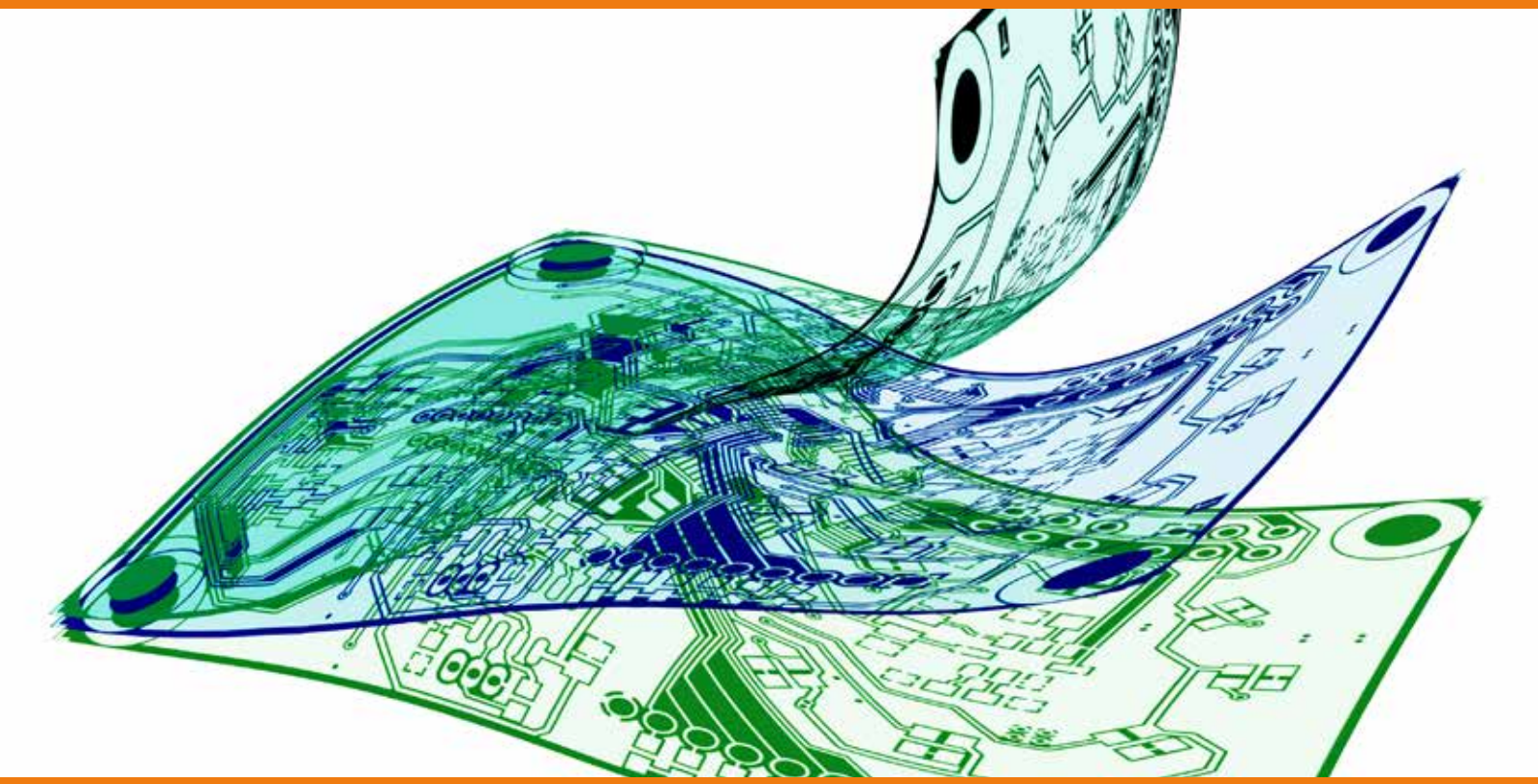
European Metrology Programme for Innovation and Research

Delivering Impact



Mass spectrometry for 3D measurements

Modern high value goods often incorporate multiple layers of different types of materials, both organic and inorganic. This complexity can cause severe measurement issues during production where any defects that occur may be buried or hard to detect. Mass spectrometry has been identified as a promising technique for revealing and identifying such problems but has lacked the resolution and traceability to be employed in this manufacturing sector.

Europe's National Measurement Institutes working together

The European Metrology Programme for Innovation and Research (EMPIR) has been developed as part of Horizon 2020, the EU Framework Programme for Research and Innovation. EMPIR funding is drawn from 28 participating EURAMET member states to support collaborative research between Measurement Institutes, academia and industry both within and outside Europe to address key metrology challenges and ensure that measurement science meets the future.

Challenge

European industry is developing 'high value-added manufacturing' which uses advanced techniques to generate a wide range of products which often contain novel characteristics such as 3D architecture, smart optical films or advanced coatings. These features, along with the rapidly expanding library of materials used, have caused severe measurement issues. One problem for multi-layered technologies are 'inclusions', or contaminants, at buried interfaces, generating defects which can compromise a product's lifetime or efficacy.

Chemical imaging using 'Time-of-Flight Secondary Ion Mass Spectrometry' (ToF-SIMS) is a promising technique for revealing and identifying these problems but a number of issues need to be addressed before it can be routinely used. ToF-SIMS typically removes material from a target by bombarding it with charged particles in a process termed 'ion beam etching'. The mass of these liberated molecules can then be determined based on how fast they move through an electric field. The current state-of-the-art mass resolution and mass accuracy, which is the ability of an instrument to resolve different chemistries, is 10 000 and greater than 30 parts-per-millions (ppm) respectively, which is insufficient to identify unknown contaminants. Furthermore, for complex multicomponent samples, ToF-SIMS can suffer from insufficient mass-resolution to separate out the individual components and insufficient mass-accuracy to chemically identify them.

Solution

The EMPIR project *Advanced 3D chemical metrology for innovative technologies* developed reliable, traceable measurement methods to characterise a novel prototype instrument, the *3D OrbiSIMS*.

The OrbiSIMS development was initiated and led by the UKs National Metrology Institution, NPL and was built through collaboration between NPL, IONTOF, ThermoFisher, GSK and academia. The instrument features a ToF-SIMS instrument in combination with an Orbitrap mass analyser which can resolve the mass of charged molecules. The incorporated dual beam technology allows it to both analyse the composition of a material and map its 3D surface features.

The project demonstrated the instruments ability for organic depth profiling to characterise buried areas, or domains, and verified a 10 to 15-fold improvement in mass resolving power compared to conventional spectrometry techniques. This increase, extending the mass resolution from 10 000-15 000 to greater than 240,000 was accompanied with a tenfold improvement in mass measurement accuracy.

Impact

IONTOF, which manufactures innovative instruments for surface analysis, is now marketing the 3D OrbiSIMS as its *Hybrid SIMS* product. IONTOF have been serving customers in the fields of microelectronics, display development, chemical and glass industry for thirty years and have over 360 different types of measurement instruments currently in use in industrial and academic laboratories worldwide. The company has continued to develop the Hybrid SIMS since the end of the project and it now has a mass accuracy of less than 3 ppm and a spatial resolution, for determining surface features, of less than 50 nanometres.

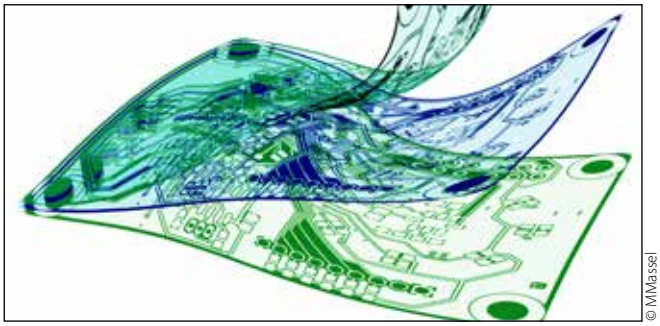
The new instrument, with accuracy levels backed by the UK's National Measurement Institute, NPL, can resolve distinct features even in highly complex organic samples, such as Organic Light Emitting Diodes (OLEDs) or biological tissue. Furthermore, the measurement of a molecule's mass by the analyser is independent of sample topography, or charging, improving the robustness of the measurement and increasing user confidence in the correct identification of a compound. For the first time chemical imaging has has the necessary resolution and sensitivity to characterise high-value manufacturing products.

Mass spectrometry for 3D measurements

The EMPIR project *Advanced 3D chemical metrology for innovative technologies* addressed the demand from industry for improved 3D chemical measurements at the micro and nano-scale. This is particularly important for the high value manufacturing sector, that experience severe measurement issues when attempting to detect defects in products which are often multi-layered and composed of both organic and inorganic materials.

The project consortium developed the first ever range of micro- and nano-structured reference materials containing different distinct chemical areas or 'domains'. Using new methodology Atom probe tomography, 3D mass spectroscopy and grazing incidence x-ray fluorescence (GIXRF) techniques were assessed for suitability to analyse complex, 3D materials.

These improved measurement capabilities will help underpin manufacturing across a range of industries where the chemical composition of devices can have a great effect on product performance and lifetime.



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Ian Gilmore

NPL, UK
+44 20 8943 6922 | Ian.Gilmore@npl.co.uk

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